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# Improvement of ternary gypsum-based composite resistance to a humid environment

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**Abstract.** The paper describes the behavior of ternary gypsum-based composites, especially the changes of properties caused by the influence of moisture. The ternary gypsum-based mortar, composed of gypsum, hydrated lime, different types of pozzolan (silica fume, granular blast slag, and brick powder), and standard sand was investigated. Pozzolans in the mixture react with hydraulic lime and form CSH phases in the material. The presence of these phases improves the resistance of gypsum against the moisture. The way of samples' storage affects the development of materials structure and properties of gypsum-based mortars. Therefore, samples were stored in two different environments (laboratory conditions and underwater) for a period of 360 days. The basic physical and mechanical properties of samples stored in the laboratory environment and under the water are compared. The comparison of the TG curves, based on the samples of different ages was carried out. The storage in the water has the most significant impact on the properties of all materials, especially on mechanical properties. While the strength of pure gypsum mortar, stored in the water, decreased significantly, and after one year in the water environment, the samples were nearly destroyed, the samples with pozzolan admixtures revealed the improvement of mechanical properties, when stored in water.

## 1. Introduction

Gypsum, as one of the historically oldest building materials, is used mainly for interior plasters. In the past, gypsum was used primarily for interior stucco decoration, but in the last decades, its significance rose due to its application in gypsum boards. Since gypsum contains a large quantity of chemically bound water, it is an excellent fire-protection material. As state-of-the-art demands on fire safety steadily increase, this increases the significance of gypsum as a fire-protection material [1-4].

However, plaster application was limited, insofar as it could not be used in areas with relatively high air humidity. Plaster is losing its strength in a humid environment, as well as it is partially soluble [5].

For the above reasons, it is an attempt to improve the properties of gypsum in a humid environment. Many authors are describing the increased gypsum resistance to moisture in case of the use of pozzolanic admixtures. For more active performance, pozzolans require the presence of calcium hydrate ( $\text{Ca}(\text{OH})_2$ ) or portland cement as their addition increases the mixture pH, allowing the so-called pozzolanic reaction. In mixtures without alkaline additives, pozzolans would act only as an inert aggregate [6, 7]. The application of pozzolan in the gypsum binder is a logical solution since the properties of both components (gypsum, lime) in the binder are complementary. While the lime shrinks during setting, the gypsum slightly expands [8], the setting of lime is slow, the gypsum sets quickly, and the workability of the gypsum-lime mixture is better.



The paper describes the effect of the application of pozzolans admixture (silica fume, slag, and brick powder) on the properties of the gypsum binder. The influence on the physical and mechanical properties of these composite materials stored in water was monitored up to 360 days of hydration.

## 2. Materials and experimental details

The used silica fume (SF) consists of more than 90 mass % of amorphous phases, the slag consists of more than 33 mass % of amorphous phases, and the brick powder (BP) consists of more than 45 mass % of amorphous phases. Hydrated lime labeled as CL-90-S (Carneuse CR, Czech Republic) was selected to act as an activator of a pozzolanic reaction. For comparison also reference admixture was prepared without pozzolan and lime (labeled as REF).

The composition of each mixture was designed with the requirement for the compatibility of the achieved results. Therefore, the dosage of pozzolan was designed according to the amount of silicon in the pozzolan. All mixtures contain the same amount of reactive phase and alkali activator. Therefore we can compare the measurement results. The rate of the physical quantity of calcium in lime hydrate and of silicon in amorphous  $\text{SiO}_2$  contained in the pozzolan is equal to 1.5. This value was proposed based on previous experiments [9]. In case this rate value is lower, the calcium hydrate would be quickly exploited, and no products of pozzolan hydration would form in the mixture. On the other hand, if the rate value is higher, some mixtures might not fulfill the condition of proportional representation of plaster.

The ratio of ternary gypsum-based binders and sand was 1:2. The amount of water in the mixture was determined by the flow test. The overflow was established to be between 185 to 190 mm concerning the workability of mortars. The amount of water is calculated as a ratio between the mass of water and the mass of the ternary binder in the mixture. The alkaline activator (lime hydrate) was added in the same amount in all mixtures. The mixture marked as REF is the reference mortar, prepared from gypsum without lime and pozzolan. Detailed compositions of mixtures are summarized in Table 1.

**Table 1.** Composition of the studied mixtures.

Mixture (pozzolan)	Gypsum [% wt]	Lime [% wt]	Pozzolan [% wt]	Sand [g]	Water-binder ratio
BP (brick powder)	73.2	10.0	16.8	1350	0.78
SF (silica fume)	81.5	10.0	8.5	1350	0.82
GBS (granulated blast slag)	81.3	10.0	8.7	1350	0.78
REF	100	0	0	1350	0.90

The samples in the shape of prisms (160×40×40 mm) were prepared from the designed mixtures. All samples were stored in laboratory conditions for 24 hours. After this time, test samples and part of reference samples were set in the water, and the second part of reference samples (labeled as REF-L) was kept in the laboratory conditions (atmospheric air, constant temperature of 25 °C).

## 3. Experimental methods

The measurements were realized, in order to find whether pozzolan admixtures were able to compensate for the decrease of strength caused by moisture. The samples were dried before testing to the constant weight at 50 °C.

Bulk density  $\rho_v$  [ $\text{kg/m}^3$ ] was determined by the gravimetric method from sample dimensions and mass of prisms.

The compressive strength was measured according to the procedure described in the technical standard EN 1015 [10]. For the measurement, three prismatic samples for each environment and hydration time were prepared. The measurements of the compressive strength were done on the halves of broken prisms with a loading area of about 1600 mm<sup>2</sup>. The uncertainty of the testing method was 1.4 %.

Simultaneous thermal analysis consisting of differential scanning calorimetry (DSC) and thermogravimetry (TG) was used to find and quantify hydration products of all studied samples. This method analyzes heat effects associated with phase transitions or chemical reactions as a function of temperature in a chosen atmosphere (inert or reactive). The experiments were done in the temperature range from 25 to 1000 °C in a nitrogen atmosphere with a flow rate of 40 mL min<sup>-1</sup>. The heating rate was 10 °C min<sup>-1</sup>.

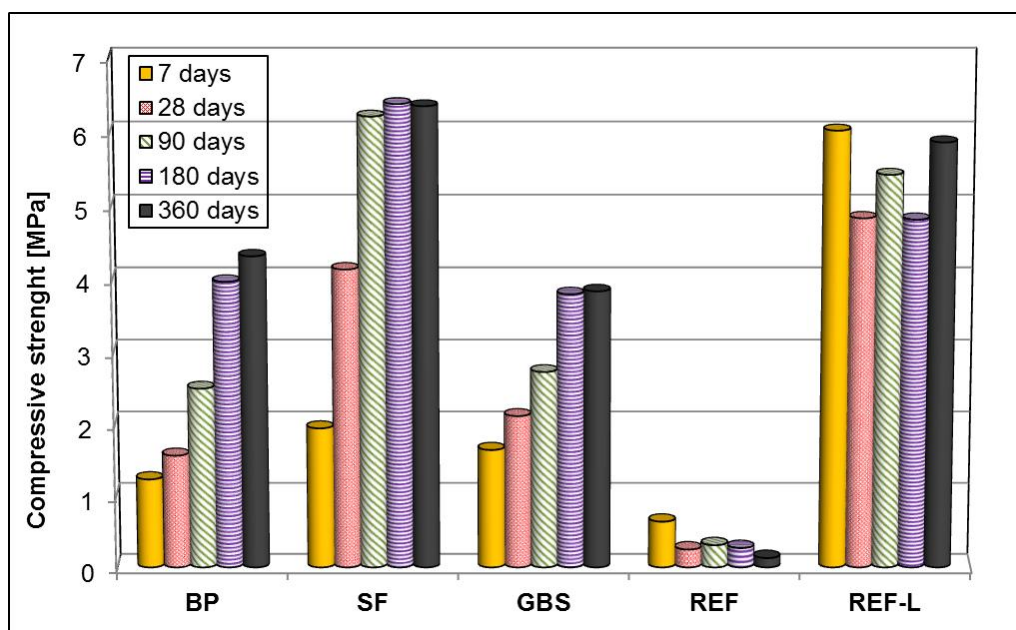
Changes in the characteristics of the samples were monitored up to 360 days of hydration.

#### 4. Results and discussion

Compressive strength up to 360 days of hydration and values of bulk density at the age of 28 days are given in Table 2 and Figure 1. The bulk density of the samples with pozzolan is as high as those of reference samples.

**Table 2.** Values of bulk density and compressive strength up to 360 days of hydration.

Mixture	Bulk density 28 days [kg/m <sup>3</sup> ]	Compressive strength [MPa]				
		7 days	28 days	90 days	180 days	360 days
<b>BP</b>	1520.1	1.23	1.56	2.49	3.96	4.31
<b>SF</b>	1698.5	1.94	4.13	6.21	6.38	6.35
<b>GBS</b>	1747.1	1.64	2.11	2.72	3.79	3.83
<b>REF</b>	1511.2	0.64	0.25	0.31	0.27	0.13
<b>REF-L</b>	1458.8	6.02	4.83	5.42	4.81	5.86

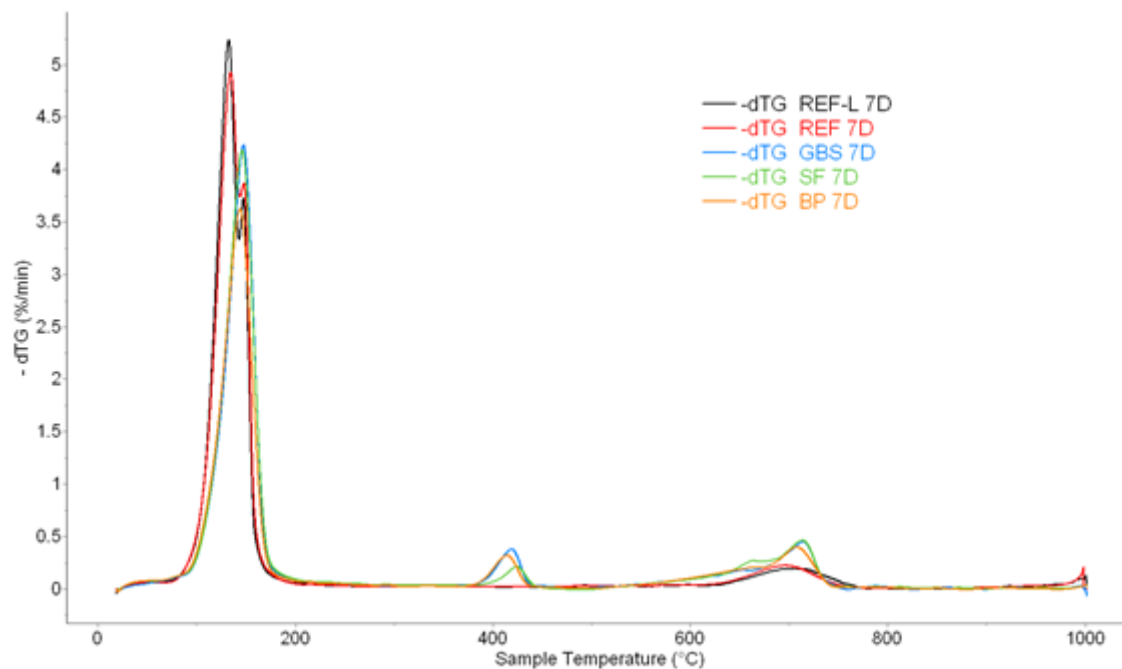


**Figure 1.** Compressive strength of studied samples.

Results of compression strengths confirmed typical gypsum behavior, where effects of mechanical properties of samples set in humid environment reach very low values while samples set in a laboratory environment (REF-L) reach high values. In samples with pozzolan set in the humid environment, a gradual compression strength improvement with the hold time in the water was evident. This trend can be explained by gradually terminating a pozzolanic reaction.

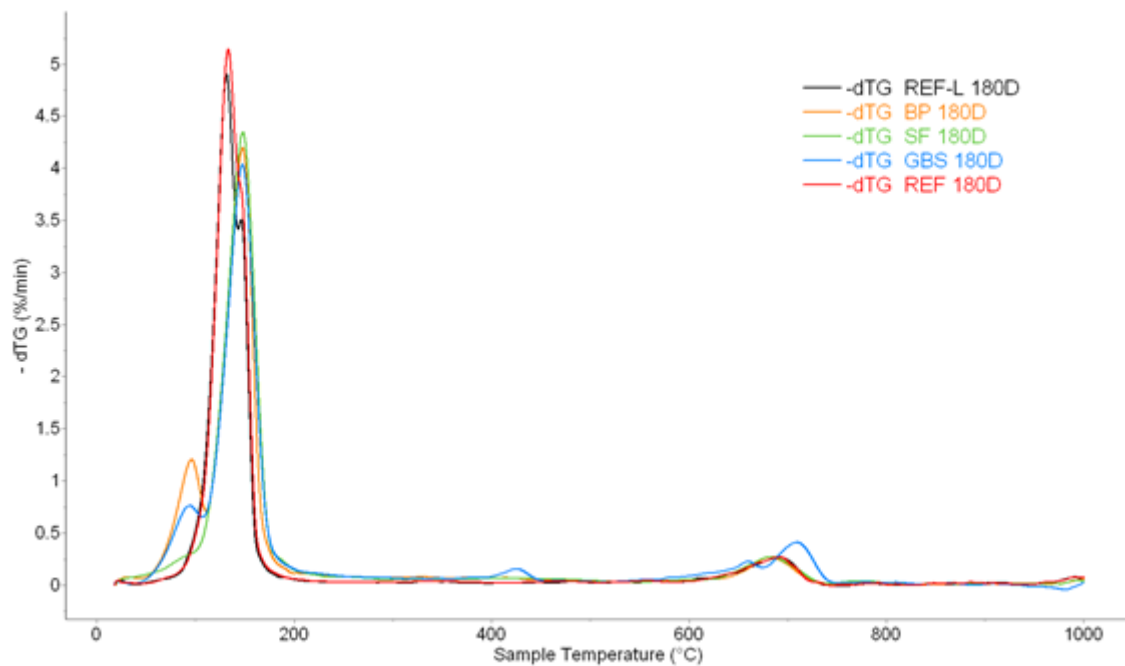
We can observe that the highest compressive strength have samples with silica fume (SF). These samples also have the highest increase of compressive strength in time, and the compressive strength increased more than three times in the period from 7 to 90 days. Samples with silica fume reached after 90 days higher strengths than reference samples (REF-L) set in laboratory conditions.

Comparison of DTG curves for samples with pozzolanic admixtures and samples REF set in the water during hydration time of 7, 180, and 360 days is shown in Figures 2-4. Figure 5 is a graphical representation of mass defects of individual samples during three temperature intervals depending on the time they were set in the water.

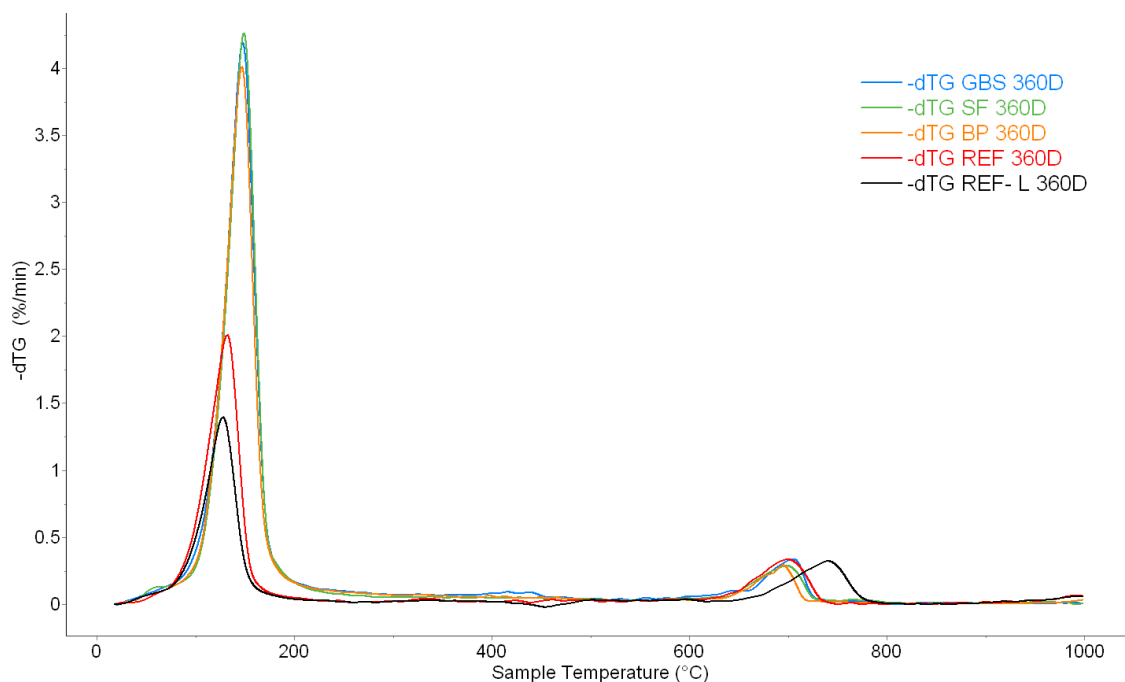


**Figure 2.** Comparison of DTG curves for samples set in the water in the 7 days.

In thermogravimetry curves of individual binder samples, four visible changes of the mass defect can be detected. At the temperature of 30-120 °C, the residual humidity is released, and CSH phase is disintegrated. Under the temperature of 120-230°C, gypsum is dehydrating into hemihydrate and anhydrite. Under the temperature of 380-460°C, portlandite decomposes into calcium oxide and water, and under the temperature 650-800°C the  $\text{CaCO}_3$  disintegrates into calcium oxide and carbon dioxide.



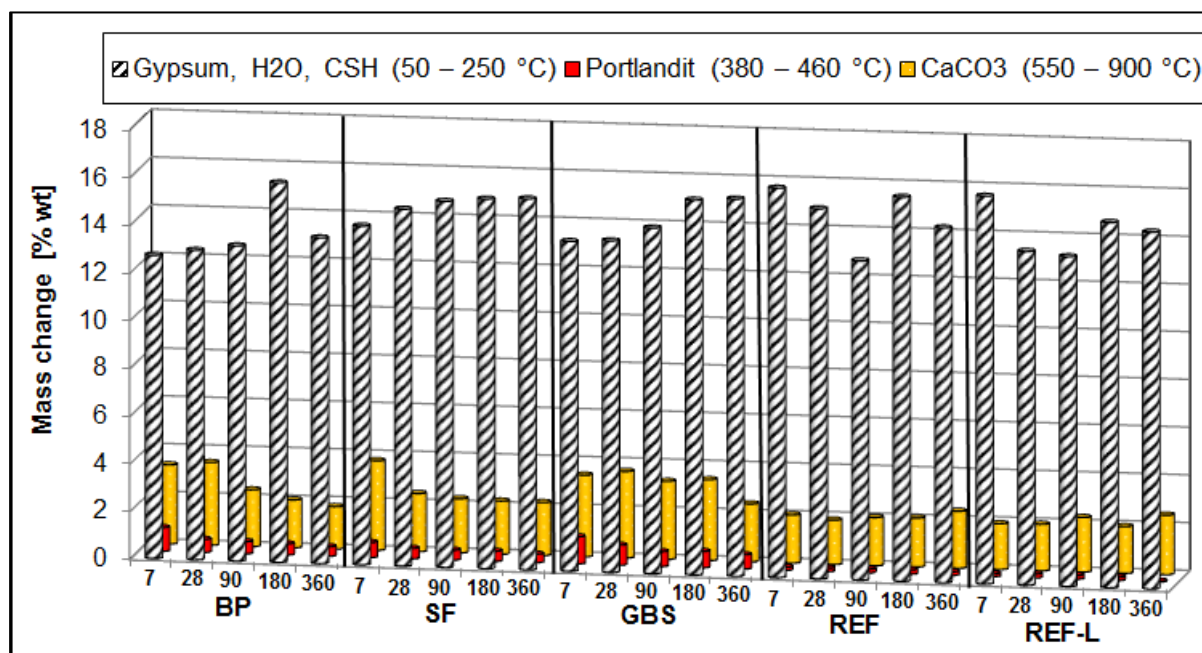
**Figure 3.** Comparison of DTG curves for samples set in the water in the 180 days.



**Figure 4.** Comparison of DTG curves for samples set in the water in the 360 days.

As is seen in Figures 2-5, after 7 days of hydration, portlandite was found in all samples with pozzolane set in the water, which is subsequently consumed by pozzolanic reaction and after 180 days is traceable in small quantity only in samples with granulated blast slag (GBS). After 360 days, portlandite is no longer present in any of the samples. BP and SF samples contain the portlandite traceable within 28 days of hydration. Mass defects within the range of temperatures (380-460 °C) in longer time intervals probably are not related to portlandite disintegration.





**Figure 5.** Mass changes of samples in 3 temperature intervals depending on time in the water.

A slow pozzolanic reaction in the sample with pozzolanic admixture set in the water is causing consumption of portlandite and further phases for the creation of CSH phases, which is proved by increasing mass defect within the range of temperatures 50-250 °C. By consumption of portlandite during the hydration process, it cannot participate in the carbonation process. It is proved by the quantity of calcite, which is almost not growing after 7 days of hydration.

From the results of the thermal analysis, it is obvious that the mass changes in the reference samples stored in both water and laboratory conditions do not show significant changes or tendencies during the storage period.

## 5. Conclusions

The ternary gypsum-based samples were prepared from the gypsum, lime, and pozzolan (brick powder, silica fume, granulated blast slag), and the reference sample was composed only of gypsum. The samples with pozzolan were stored in the water environment, and reference samples were adjusted in both the water environment and the air in laboratory conditions. Changes in the characteristics (mass change, changes in DTA curves, and change of compressive strength) of the samples were monitored from 7 up to 360 days of hydration.

Application of pozzolanic admixture in the gypsum-based binder is significantly improving material properties (i.e., mechanical properties) of test specimens of binders. It is evident from results that the application of pozzolan caused in the samples products pozzolanic reaction, which has a positive impact on water transport properties, caused by latent reaction with portlandite ( $\text{Ca}(\text{OH})_2$ ).

Puzzolan modified ternary binders have a higher bulk density than reference samples. The remarkable difference was found in compressive strengths in samples with pozzolanic admixtures set in the water where reference samples had very low compressive strengths, and pozzolanic samples had much higher compressive strength. The best results were reached in the silica fume admixture, where after 90 days of the setting of samples in the water was reached higher compressive strength than in reference samples from laboratory conditions. This improvement was reached due to the highest pozzolanic reactivity of microsilica as proved results of the thermal (DTG) analysis.

The presence of a pozzolanic reaction was proved by loss of portlandite and growth of the number of C-S-H hydration phases during the hydration time leading to a slowing of carbonation. Changes in the chemical composition of these phases were proven by DTG analysis

Therefore the resistance of the ternary materials against water is better than the resistance of the materials without pozzolans, which might contribute to a wider application of gypsum-based products.

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